USGS
science for a changing world

Prepared in collaboration with the Missouri Department of Conservation

Concentrations of Elements in Fish Fillets, Fish Muscle Plugs, and Crayfish from the 2011 Missouri Department of Conservation General Contaminant Monitoring Program

Open-File Report 2012–1268

U.S. Department of the Interior
U.S. Geological Survey

Concentrations of Elements in Fish Fillets, Fish Muscle Plugs, and Crayfish from the 2011 Missouri Department of Conservation General Contaminant Monitoring Program

By Thomas W. May, Michael J. Walther, William G. Brumbaugh, and Michael J. McKee

Prepared in collaboration with the Missouri Department of Conservation

Open-File Report 2012–1268

U.S. Department of the Interior
U.S. Geological Survey

U.S. Department of the Interior
KEN SALAZAR, Secretary

U.S. Geological Survey
Marcia K. McNutt, Director

U.S. Geological Survey, Reston, Virginia: 2013

For more information on the USGS—the Federal source for science about the Earth, its natural and living resources, natural hazards, and the environment, visit http://www.usgs.gov or call 1–888–ASK–USGS.

For an overview of USGS information products, including maps, imagery, and publications, visit http://www.usgs.gov/pubprod

To order this and other USGS information products, visit http://store.usgs.gov

Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Although this information product, for the most part, is in the public domain, it also may contain copyrighted materials as noted in the text. Permission to reproduce copyrighted items must be secured from the copyright owner.

Suggested citation:
May, T.W., Walther, M.J., Brumbaugh, W.G., and McKee, M.J., 2013, Concentrations of elements in fish fillets, fish muscle plugs, and crayfish from the 2011 Missouri Department of Conservation general contaminant monitoring program: U.S. Geological Survey Open-File Report 2012–1268, 12 p.

Contents

Abstract....1
Introduction....1
Sampling History....1
Methods....2
Sites and Sample Collection....2
Homogenization and Lyophilization....2
Chemical Preparation....2
Instrumental Analysis....2
Quality Control....2
Results....2
Quality-Control Results....11
References Cited....12

Tables

1. Missouri collection sites for fish fillets, fish muscle plugs, and crayfish from the 2011 Missouri Department of Conservation General Contaminant Monitoring Program....3
2. Percent moisture and dry weight concentrations of calcium, zinc, cadmium, lead, and lead/calcium molar ratios in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples....4
3. Percent moisture and wet weight concentrations of calcium, zinc, cadmium, and lead in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples....6
4. Concentrations of total mercury in fish fillets, fish muscle plugs, and crayfish tissues....8

Conversion Factors

Multiply	By	To obtain
	Length	
millimeter (mm)	0.03937	inch (in.)
micrometer	0.0000393	inch (in.)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
milliliter (mL)	.034	ounce, fluid (fl. oz)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
milligram (mg)	.000035	ounce, avoirdupois (oz)
microgram (µg)	0.000000035	ounce, avoirdupois (oz)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:
°F=(1.8×°C)+32

Concentrations of chemical constituents in water are given in nanograms per milliliter (ng/mL).

Concentrations of chemical constituents in solid materials are given in either micrograms per gram (µg/g) dry weight or µg/g wet weight.

Concentrations of Elements in Fish Fillets, Fish Muscle Plugs, and Crayfish from the 2011 Missouri Department of Conservation General Contaminant Monitoring Program

By Thomas W. May[1], Michael W. Walther[1], William G. Brumbaugh[1], and Michael J. McKee[2]

Abstract

This report presents the results of a contaminant monitoring survey conducted annually by the Missouri Department of Conservation to examine the levels of selected elemental contaminants in fish fillets, fish muscle plugs, and crayfish. Fillet samples of yellow bullhead (*Ameiurus natalis*), golden redhorse (*Moxostoma erythrurum*), longear sunfish (*Lepomis megalotis*), and channel catfish (*Ictalurus punctatus*) were collected from six sites as part of the Missouri Department of Conservation's Fish Contaminant Monitoring Program. Fish dorsal muscle plugs were collected from largemouth bass (*Micropterus salmoides*) at eight of the sites, and crayfish from two sites. Following preparation and analysis of the samples, highlights of the data were as follows: cadmium and lead residues were most elevated in crayfish tissue samples from the Big River at Cherokee Landing, with 1 to 8 micrograms per gram dry weight and 22 to 45 micrograms per gram dry weight, respectively. Some dorsal muscle plugs from largemouth bass collected from Clearwater Lake, Lake St. Louis, Noblett Lake, Hazel Creek Lake, and Harrison County Lake contained mercury residues (1.7 to 4.7 micrograms per gram dry weight) that exceeded the U.S. Environmental Protection Agency Water Quality Criterion of 1.5 micrograms per gram dry weight of fish tissue (equivalent to 0.30 micrograms per gram wet weight).

Introduction

The Missouri Department of Conservation (MDC) initiated long-term statewide fish monitoring of environmental contaminants in 1984 with the objective of selecting predator and bottom dwelling species annually from each of 20 to 30 lakes and streams across Missouri to characterize concentrations of targeted metals and other chemical contaminants. Actual sites monitored each year vary based on data needs, budgets, and personnel resources. Emphasis is on human health effects from recreational and sport fishing, so study components include fish fillets, composite fillet samples, and sample replication at each site. The MDC has requested the assistance of the U.S. Geological Survey (USGS) for this monitoring program because of past experience with aquatic biota monitoring projects and expertise in the preparation and analysis of aquatic biota for elemental contaminants. For more detailed information on the overall study design or specific sample information, please contact the Missouri Department of Conservation in Columbia, Missouri (*http://mdc.mo.gov/regions/central*).

Sampling History

A shipment of 27 whole-body fish, 25 fish fillets, 48 fish muscle plugs, and 37 crayfish tissues was received by USGS personnel on December 7, 2011. The samples included skinless fillets collected from yellow bullhead, golden redhorse, longear sunfish, and channel catfish to reflect the form of fish tissue customarily consumed by anglers. Dorsal fish muscle plugs were collected from largemouth bass for purposes of assessing mercury accumulation. Crayfish tissues were collected for assessing lead accumulation. Upon receipt, the shipment was assigned USGS batch number 1974 and sample identifications (IDs) 55878-55987. All samples had been stored frozen at -20 degrees Celsius (º C) since collection at the MDC's Resource Science Center in Columbia, Missouri, and were delivered by MDC personnel. Requested analyses included zinc (Zn), cadmium (Cd), mercury (Hg), and lead (Pb). Calcium (Ca) also was analyzed in an effort to explain Pb variability (Schmitt and Finger, 1987). Fish fillet samples were variable in the way they were obtained, and the fillet technique may have resulted in different amounts of tissue extracted and, thus, may have varying amounts of calcium-rich bone fragments that can be comparatively high in Pb.

[1]U.S. Geological Survey

[2]Missouri Department of Conservation

Methods

Sites and Sample Collection

In 2011, 16 sites (table 1) were selected for sampling as part of the MDC General Contaminant Monitoring Program (GCMP). The 2011 collection consisted of yellow bullhead (*Ameiurus natalis*), golden redhorse (*Moxostoma erythrurum*), longear sunfish (*Lepomis megalotis*), and channel catfish (*Ictalurus punctatus*). Besides fish fillet samples collected in 2011, fish dorsal muscle plugs were collected from largemouth bass (*Micropterus salmoides*), and tissues [tail shell, tail-meat, and carcass (crayfish remains after tail meat was removed or extracted)] were collected from crayfish.

Homogenization and Lyophilization

GCMP fillet samples were chopped and minced with a titanium meat cleaver on a cleaned polypropylene cutting board and then lyophilized. Fish muscle plugs and crayfish tissues (tail meat and non tail meat) were lyophilized as received. After lyophilization, fish fillet samples were hand-kneaded in a plastic (polyethylene) bag or crushed with a rolling pin in a plastic bag to achieve a coarse powder consistency, whereas crayfish tissues were placed in a glass vial and ground with a glass rod. All dried and ground products were stored at room temperature in a 40-milliliter (mL) glass vial in a nitrogen desiccator.

Chemical Preparation

To prepare fish samples for analysis of Ca, Zn, Cd, and Pb, a dried sample of approximately 0.25 grams (g) was heated with 6mL of nitric acid in a sealed high-pressure Teflon® vessel in a laboratory microwave oven. The cooled digestate liquid was transferred into a 125-mL polyethylene bottle with ultrapure water [specific resistance greater than (>) 10 megOhms per centimeter (megOhm/cm)] and diluted to a final weight of 101.5 g. Final acid matrix was 6 percent by volume concentrated nitric acid. For the determination of Hg in fish samples, there was no chemical preparation (digestion) because the dried sample was decomposed thermally during instrumental analysis (see Instrumental Analysis section).

Instrumental Analysis

Calcium, Zn, Cd, and Pb analyses were conducted using a PE/SCIEX Elan 6000® inductively coupled plasma-mass spectrometer (ICP-MS), which was set up in "Standard Mode" and optimized according to the manufacturer's specifications. Samples were delivered automatically to the ICP-MS by means of a software-controlled CETAC ASD-500 autosampler/autodiluter system. All sample digestates were analyzed with a 10-fold predilution by autodiluter.

The ICP-MS quantitative method was designed to determine the following masses: ^{44}Ca and ^{48}Ca, ^{60}Zn and ^{68}Zn, ^{111}Cd and ^{114}Cd, and Pb as the sum of three masses (^{206}Pb+^{207}Pb+^{208}Pb). The internal standards were scandium (Sc) at 10 nanograms per milliliter (ng/mL), rhodium (Rh; 10 ng/mL), and bismuth (Bi; 10 ng/mL), which were metered into the sample line via peristaltic pump. Calibration standards for analyses were as follows: Ca - 2500, 5000, and 10,000 ng/mL; Zn - 75, 150, and 300 ng/mL; Pb - 5, 10, 20, and 40 ng/mL; and Cd - 1.5, 3.0, 6.0, and 12 ng/mL. During the actual analysis, any digestate concentration greater than the upper calibration standard for any element was automatically diluted 10-fold serially until its concentration was below this level. Where multiple masses for an element were measured, the concentration reported was based on the mass exhibiting least interferences, which were as follows: ^{44}Ca, ^{60}Zn, ^{114}Cd , and Pb as the sum of three masses ^{206}Pb+^{207}Pb+^{208}Pb).

Mercury was determined with a Milestone DMA-80 analyzer equipped with an automated sample carousel. With this method, a dried fish sample [40 to 60 milligrams (mg)] was combusted in a stream of oxygen. All Hg in the sample was volatilized and trapped by amalgamation on a gold substrate, and was thermally desorbed and quantitated by atomic absorption spectrophotometry (U. S. Environmental Protection Agency, 1998).

Quality Control

The samples were digested and analyzed in two groups or batches for Ca, Zn, Cd, and Pb, and four batches for Hg. The quality-control incorporated in the digestion stage of the samples (for subsequent ICP-MS analysis) included digestion blanks, reference materials, replicates, and spikes. For the determination of Ca, Zn, Cd, and Pb by ICP-MS, instrumental quality control included calibration checks, laboratory control solutions, duplicate digestate analysis, analysis spikes, and interference checks (dilution percent difference and a synthetic interference solution). Quality control for Hg included blanks, independent calibration verification checks, replicates, pre-combustion spikes, and tissue reference materials. All quality-control results were tabulated to provide an overview of quality assurance and to facilitate interpretation.

Results

Percent moisture, concentrations in micrograms per gram (µg/g) dry weight of Ca, Zn, Cd, and Pb, and molar ratios (fillet samples only) of [Pb/Ca] (1×10^{-6}) for the fish fillet and crayfish tissues are presented in table 2. To facilitate comparison with any regulatory guidelines, concentrations of Ca, Zn, Cd, and Pb are presented for these matrices in micrograms per gram wet weight in table 3. Concentrations of total mercury in micrograms per gram dry and wet weight in fish fillets, fish

Table 1. Missouri collection sites for fish fillets, fish muscle plugs, and crayfish from the 2011 Missouri Department of Conservation General Contaminant Monitoring Program.

Species and Common Name	Sites	Sample Type
Ameiurus natalis	Mound Branch at Paxton, Missouri	Fillet
Yellow Bullhead		
Moxostoma erythrurum	Big River at House Springs	Fillet
Golden Redhorse	Meramec River at Allenton Access	Fillet
	Meramec River at Route 66 State Park	Fillet
Lepomis megalotis	Meramec River at Allenton Access	Fillet
Longear Sunfish	Big River at House Springs	Fillet
	Meramec River at Route 66 State Park	Fillet
Ictalurus punctatus	Wakonda State Park at Agate Lake	Fillet
Channel Catfish	Wakonda State Park at Wakonda Lake	Fillet
Micropterus salmoides	Lake of the Ozarks at Niangua Arm	Plug
Largemouth Bass	Longview Lake	Plug
	Stockton Lake at Big Sac Arm	Plug
	Clearwater Lake	Plug
	Lake St. Louis	Plug
	Noblett Lake	Plug
	Hazel Creek Lake	Plug
	Harrison County Lake	Plug
Crayfish species	Big River at Cherokee Landing	Tail meat
		Non tail meat
	Shoal Creek 0.5 mile North of I-44 Bridge	Tail meat
		Non tail meat
		Tail shell

muscle plugs, and crayfish tissues are presented in table 4. For this report all sample and quality-control data are discussed in terms of dry weight results only.

Calcium concentrations were variable among fillet samples; concentrations were generally within a factor of two or three among golden redhorse, but increased to a factor of seven in longear sunfish (table 2). Such variation presumably was because of variable bone content in the fillets and undoubtedly was affected by significant variations in fillet preparation techniques as well as by the fish species. For example, catostomids possess numerous tiny intermuscular bones. Accordingly, fillets from some species (golden redhorse and longear sunfish) contained higher Ca concentrations than other species (catfish). Although Ca normally is not a target analyte, fillet Ca concentrations are useful when target analytes include those that markedly accumulate in bone (for example, Pb). Thus, measurements of Ca can help explain high variation in Pb concentrations for individual samples that included variable amounts of Ca-rich tissue (Schmitt and Finger, 1987). Concentrations of Ca in crayfish tail-meat were similar to those of fish fillets, but concentrations in crayfish carcass remains and shell samples were some 100 fold higher.

Zinc concentrations were quite uniform in fillet samples among the different species of fish, ranging from 20 to 31 µg/g dry weight. Concentrations of Zn (µg/g dry weight) in crayfish tissues were lowest in tail shell samples (17 to 25), but three to six fold higher in tail meat and carcass samples (71 to 134). Cadmium concentrations (µg/g dry weight) were less than 0.1 in most fish fillet samples (72 percent of sample set) and ranged from 0.1 to 0.26 in remaining fillet samples. Cadmium

Table 2. Percent moisture and dry weight concentrations of calcium, zinc, cadmium, lead, and lead/calcium molar ratios in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples.

[USGS, U.S. Geological Survey; ID, identification; MDC, Missouri Department of Conservation; Ca, calcium; µg/g, micrograms per gram dry weight; Zn, zinc; Cd, cadmium; Pb, lead; <, less than; bold and italicized values are greater than the method detection limit but less than the method quantitation limit and have higher uncertainty]

USGS ID	MDC ID	Biota common name	Matrix	Percent moisture	Ca µg/g	Zn µg/g	Cd µg/g	Pb µg/g	Pb/Ca molar ratio ($1X10^{-6}$)
55881	2011-723-240-1	Yellow Bullhead	Fish fillet	81.9	776	31.3	<0.012	0.10	25
55878	2011-056-062-1	Golden Redhorse	Fish fillet	81.0	4,960	20.9	***0.034***	1.50	59
55879	2011-056-062-2	Golden Redhorse	Fish fillet	81.5	4,690	23.8	0.04	1.39	57
55880	2011-056-062-3	Golden Redhorse	Fish fillet	79.9	3,170	21.0	***0.025***	0.82	50
55882	2011-724-062-1	Golden Redhorse	Fish fillet	80.7	3,230	24.8	***0.032***	0.12	7.2
55883	2011-724-062-2	Golden Redhorse	Fish fillet	80.5	1,340	21.2	0.075	0.037	5.3
55884	2011-724-062-3	Golden Redhorse	Fish fillet	80.1	2,270	24.0	0.26	0.087	7.4
55885	2011-725-062-1	Golden Redhorse	Fish fillet	80.8	4,640	25.1	0.19	0.37	15
55886	2011-725-062-2	Golden Redhorse	Fish fillet	81.5	3,050	21.2	0.10	0.21	13
55887	2011-725-062-3	Golden Redhorse	Fish fillet	80.8	3,190	22.3	0.093	0.21	13
55888	2011-724-420-1	Longear Sunfish	Fish fillet	81.4	818	20.4	0.047	***0.020***	4.7
55889	2011-724-420-2	Longear Sunfish	Fish fillet	82.8	1,440	23.6	0.11	***0.023***	3.1
55890	2011-724-420-3	Longear Sunfish	Fish fillet	81.1	1,820	22.8	0.14	0.057	6.1
55891	2011-056-420-1	Longear Sunfish	Fish fillet	81.2	3,910	29.8	***0.038***	0.81	40
55892	2011-056-420-2	Longear Sunfish	Fish fillet	80.2	6,260	26.3	***0.028***	1.11	34
55893	2011-056-420-3	Longear Sunfish	Fish fillet	78.9	5,550	25.7	0.041	1.12	39
55900	2011-725-420-1	Longear Sunfish	Fish fillet	81.5	1,920	22.6	0.14	0.19	19
55901	2011-725-420-2	Longear Sunfish	Fish fillet	81.8	1,690	24.2	0.15	0.18	21
55902	2011-725-420-3	Longear Sunfish	Fish fillet	81.5	834	24.5	0.066	0.052	12
55894	2011-459-230-1	Channel Catfish	Fish fillet	83.8	590	27.4	<0.012	0.069	23
55895	2011-459-230-2	Channel Catfish	Fish fillet	84.5	604	31.4	<0.012	0.033	11
55896	2011-459-230-3	Channel Catfish	Fish fillet	82.7	546	26.1	<0.012	0.037	13
55897	2011-514-230-1	Channel Catfish	Fish fillet	78.1	489	20.8	<0.012	***0.023***	9.1
55898	2011-514-230-2	Channel Catfish	Fish fillet	78.3	527	24.0	<0.012	0.029	11
55899	2011-514-230-3	Channel Catfish	Fish fillet	80.0	538	24.9	<0.012	***0.018***	6.5
55952	2011-134-609-1-S	Crayfish species	Carcass[a]	73.8	247,000	70.2	4.78	21.9	17
55954	2011-134-609-2-S	Crayfish species	Carcass	71.4	166,000	93.8	5.74	29.0	34
55956	2011-134-609-3-S	Crayfish species	Carcass	71.3	115,000	115	3.03	31.8	53
55958	2011-134-609-4-S	Crayfish species	Carcass	74.4	255,000	73.8	4.03	28.9	22

Table 2. Percent moisture and dry weight concentrations of calcium, zinc, cadmium, lead, and lead/calcium molar ratios in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples.—Continued

[USGS, U.S. Geological Survey; ID, identification; MDC, Missouri Department of Conservation; Ca, calcium; µg/g, micrograms per gram dry weight; Zn, zinc; Cd, cadmium; Pb, lead; <, less than; bold and italicized values are greater than the method detection limit but less than the method quantitation limit and have higher uncertainty]

USGS ID	MDC ID	Biota common name	Matrix	Percent moisture	Ca µg/g	Zn µg/g	Cd µg/g	Pb µg/g	Pb/Ca molar ratio ($1X10^{-6}$)
55960	2011-134-609-5-S	Crayfish species	Carcass	80.0	182,000	92.2	7.79	37.5	40
55962	2011-134-609-6-S	Crayfish species	Carcass	72.1	151,000	101	5.50	40.6	52
55964	2011-134-609-7-S	Crayfish species	Carcass	77.6	173,000	77.4	5.82	27.9	31
56227	2011-134-609-M Composite[b]	Crayfish species	Tail meat	83.2[c]	3,550	116	1.06	44.9	2,450
55965	2011-726-609-1-M	Crayfish species	Tail meat	85.6	1,560	79.1	0.027	0.29	36
55966	2011-726-609-1-S	Crayfish species	Carcass	75.9	133,000	134	0.46	0.59	0.9
55967	2011-726-609-2-M	Crayfish species	Tail meat	82.6	2,370	69.1	0.02	0.27	22
55968	2011-726-609-2-S	Crayfish species	Carcass	70.1	164,000	75.6	0.40	0.72	0.8
55969	2011-726-609-3-M	Crayfish species	Tail meat	78.7	1,210	78.4	0.046	0.26	42
55970	2011-726-609-3-S	Crayfish species	Carcass	65.6	127,000	94.5	0.60	2.43	3.7
55971	2011-726-609-4-M	Crayfish species	Tail meat	79.1	1,270	76.8	0.028	0.18	27
55972	2011-726-609-4-S	Crayfish species	Carcass	68.4	114,000	99.7	0.43	2.00	3.4
55973	2011-726-609-5-M	Crayfish species	Tail meat	78.8	2,090	81.2	0.027	0.28	26
55974	2011-726-609-5-S	Crayfish species	Carcass	65.4	124,000	129	0.50	3.33	5.2
55975	2011-726-609-6-M	Crayfish species	Tail meat	85.3	2,720	85.1	0.017	0.18	13
55976	2011-726-609-6-S	Crayfish species	Carcass	73.1	172,000	83.6	0.20	0.70	0.8
55977	2011-726-609-7-M	Crayfish species	Tail meat	81.6	3,670	70.7	0.028	0.17	9.0
55978	2011-726-609-7-S	Crayfish species	Carcass	67.5	128,000	112	0.31	1.10	1.7
55979	2011-726-609-8-M	Crayfish species	Tail meat	87.7	2,570	98.7	0.33	4.74	357
55980	2011-726-609-8-S	Crayfish species	Carcass	74.2	144,000	102	0.24	1.06	1.4
55981	2011-726-609-8-T	Crayfish species	Tail shell	72.2	239,000	17.2	0.029	0.69	0.6
55982	2011-726-609-9-M	Crayfish species	Tail meat	86.3	4,170	82.1	0.09	1.61	75
55983	2011-726-609-9-S	Crayfish species	Carcass	69.8	112,000	98.3	0.23	0.93	1.6
55984	2011-726-609-9-T	Crayfish species	Tail shell	68.1	165,000	24.9	0.02	0.86	1.0
55985	2011-726-609-10-M	Crayfish species	Tail meat	86.5	2,890	93.4	0.19	2.78	186
55986	2011-726-609-10-S	Crayfish species	Carcass	69.5	130,000	101	0.23	0.95	1.4
55987	2011-726-609-10-T	Crayfish species	Tail shell	68.9	228,000	20.6	0.042	1.24	1.1

[a]Carcass is defined as crayfish remains after tail meat has been removed or extracted.

[b]Because of insufficient biomass in submitted individual samples, this sample is a composite of seven crayfish tail meat samples collected from the Big River at Cherokee Landing (55951, 55953, 55955, 55957, 55959, 55961, 55963).

[c]Average moisture for all crayfish tail meat samples used as moisture value for this tail meat composite.

Table 3. Percent moisture and wet weight concentrations of calcium, zinc, cadmium, and lead in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples.

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; ID, identification; Ca, calcium; µg/g, micrograms per gram wet weight; Zn, zinc; Cd, cadmium; Pb, lead; <, less than; bold and italicized values are greater than the method detection limit but less than the method quantitation limit and have higher uncertainty]

USGS ID	MDC ID	Biota common name	Matrix	Percent moisture	Ca µg/g	Zn µg/g	Cd µg/g	Pb µg/g
55878	2011-056-062-1	Golden Redhorse	Fish fillet	81.0	941	4.0	***0.006***	0.28
55879	2011-056-062-2	Golden Redhorse	Fish fillet	81.5	869	4.4	0.007	0.26
55880	2011-056-062-3	Golden Redhorse	Fish fillet	79.9	638	4.2	***0.005***	0.17
55881	2011-723-240-1	Yellow Bullhead	Fish fillet	81.9	140	5.7	<0.003	0.018
55882	2011-724-062-1	Golden Redhorse	Fish fillet	80.7	623	4.8	***0.006***	0.023
55883	2011-724-062-2	Golden Redhorse	Fish fillet	80.5	261	4.1	0.015	0.007
55884	2011-724-062-3	Golden Redhorse	Fish fillet	80.1	452	4.8	0.052	0.017
55885	2011-725-062-1	Golden Redhorse	Fish fillet	80.8	891	4.8	0.036	0.071
55886	2011-725-062-2	Golden Redhorse	Fish fillet	81.5	564	3.9	0.018	0.039
55887	2011-725-062-3	Golden Redhorse	Fish fillet	80.8	612	4.3	0.018	0.040
55888	2011-724-420-1	Longear Sunfish	Fish fillet	81.4	152	3.8	0.009	***0.004***
55889	2011-724-420-2	Longear Sunfish	Fish fillet	82.8	247	4.0	0.019	***0.004***
55890	2011-724-420-3	Longear Sunfish	Fish fillet	81.1	345	4.3	0.027	0.011
55891	2011-056-420-1	Longear Sunfish	Fish fillet	81.2	733	5.6	***0.007***	0.15
55892	2011-056-420-2	Longear Sunfish	Fish fillet	80.2	1,240	5.2	***0.006***	0.22
55893	2011-056-420-3	Longear Sunfish	Fish fillet	78.9	1,170	5.4	0.009	0.24
55900	2011-725-420-1	Longear Sunfish	Fish fillet	81.5	356	4.2	0.026	0.035
55901	2011-725-420-2	Longear Sunfish	Fish fillet	81.8	307	4.4	0.027	0.033
55902	2011-725-420-3	Longear Sunfish	Fish fillet	81.5	154	4.5	0.012	0.010
55894	2011-459-230-1	Channel Catfish	Fish fillet	83.8	95	4.4	<0.003	0.011
55895	2011-459-230-2	Channel Catfish	Fish fillet	84.5	93	4.9	<0.004	0.005
55896	2011-459-230-3	Channel Catfish	Fish fillet	82.7	94	4.5	<0.005	0.006
55897	2011-514-230-1	Channel Catfish	Fish fillet	78.1	107	4.6	<0.006	***0.005***
55898	2011-514-230-2	Channel Catfish	Fish fillet	78.3	115	5.2	<0.007	0.006
55899	2011-514-230-3	Channel Catfish	Fish fillet	80.0	107	5.0	<0.008	***0.004***
55952	2011-134-609-1-S	Crayfish species	Carcass[a]	73.8	64,700	18.	1.25	***5.74***
55954	2011-134-609-2-S	Crayfish species	Carcass	71.4	47,400	27.	1.64	8.29
55956	2011-134-609-3-S	Crayfish species	Carcass	71.3	33,100	33.	0.87	9.14
55958	2011-134-609-4-S	Crayfish species	Carcass	74.4	65,300	19.	1.03	7.40
55960	2011-134-609-5-S	Crayfish species	Carcass	80.0	36,400	18.	1.56	7.50

Table 3. Percent moisture and wet weight concentrations of calcium, zinc, cadmium, and lead in Missouri Department of Conservation 2011 General Contaminant Monitoring Program fish fillet, fish muscle plug, and crayfish samples.—Continued

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; ID, identification; Ca, calcium; µg/g, micrograms per gram wet weight; Zn, zinc; Cd, cadmium; Pb, lead; <, less than; bold and italicized values are greater than the method detection limit but less than the method quantitation limit and have higher uncertainty]

USGS ID	MDC ID	Biota common name	Matrix	Percent moisture	Ca µg/g	Zn µg/g	Cd µg/g	Pb µg/g
55962	2011-134-609-6-S	Crayfish species	Carcass	72.1	42,100	28.	1.53	11.3
55964	2011-134-609-7-S	Crayfish species	Carcass	77.6	38,700	17.	1.30	6.25
56227	2011-134-609-M Composite[b]	Crayfish species	Tail meat	83.2[c]	596	19.	0.18	7.54
55965	2011-726-609-1-M	Crayfish species	Tail meat	85.6	225	11.	0.004	0.042
55966	2011-726-609-1-S	Crayfish species	Carcass	75.9	32,100	32.	0.11	0.14
55967	2011-726-609-2-M	Crayfish species	Tail meat	82.6	412	12.	0.003	0.047
55968	2011-726-609-2-S	Crayfish species	Carcass	70.1	49,100	23.	0.12	0.22
55969	2011-726-609-3-M	Crayfish species	Tail meat	78.7	258	17.	0.010	0.055
55970	2011-726-609-3-S	Crayfish species	Carcass	65.6	43,700	32.	0.21	0.84
55971	2011-726-609-4-M	Crayfish species	Tail meat	79.1	266	16.	0.006	0.038
55972	2011-726-609-4-S	Crayfish species	Carcass	68.4	36,000	31.	0.14	0.63
55973	2011-726-609-5-M	Crayfish species	Tail meat	78.8	444	17.	0.006	0.059
55974	2011-726-609-5-S	Crayfish species	Carcass	65.4	42,900	45.	0.17	1.15
55975	2011-726-609-6-M	Crayfish species	Tail meat	85.3	400	13.	0.002	0.026
55976	2011-726-609-6-S	Crayfish species	Carcass	73.1	46,200	22.	0.054	0.19
55977	2011-726-609-7-M	Crayfish species	Tail meat	81.6	677	13.	0.005	0.031
55978	2011-726-609-7-S	Crayfish species	Carcass	67.5	41,500	36.	0.10	0.36
55979	2011-726-609-8-M	Crayfish species	Tail meat	87.7	317	12.	0.041	0.59
55980	2011-726-609-8-S	Crayfish species	Carcass	74.2	37,100	26.	0.062	0.27
55981	2011-726-609-8-T	Crayfish species	Tail shell	72.2	66,500	4.8	0.008	0.19
55982	2011-726-609-9-M	Crayfish species	Tail meat	86.3	570	11.	0.013	0.22
55983	2011-726-609-9-S	Crayfish species	Carcass	69.8	33,800	30.	0.069	0.28
55984	2011-726-609-9-T	Crayfish species	Tail shell	68.1	52,700	8.0	0.006	0.27
55985	2011-726-609-10-M	Crayfish species	Tail meat	86.5	389	13.	0.026	0.37
55986	2011-726-609-10-S	Crayfish species	Carcass	69.5	39,600	31.	0.070	0.29
55987	2011-726-609-10-T	Crayfish species	Tail shell	68.9	70,800	6.4	0.013	0.39

[a]Carcass is defined as crayfish remains after tail meat has been removed or extracted.

[b]Because of insufficient biomass in submitted individual samples, this sample is a composite of 7 crayfish tail meat samples collected from the Big River at Cherokee Landing (55951, 55953, 55955, 55957, 55959, 55961, 55963).

[c]Average moisture for all crayfish tail meat samples used as moisture value for this tail meat composite..

Table 4. Concentrations of total mercury in fish fillets, fish muscle plugs, and crayfish tissues.

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; Hg, mercury; µg/g, micrograms per gram; wet weight concentrations estimated assuming 80 percent moisture].

USGS ID	MDC ID	Site location	Biota common name	Matrix	Hg µg/g dry weight	Hg µg/g wet weight
55878	2011-056-062-1	Big River at House Springs	Golden Redhorse	Fish fillet	0.21	0.042
55879	2011-056-062-2	Big River at House Springs	Golden Redhorse	Fish fillet	0.29	0.059
55880	2011-056-062-3	Big River at House Springs	Golden Redhorse	Fish fillet	0.18	0.036
55881	2011-723-240-1	Mound Branch at Paxton, Missouri	Yellow Bullhead	Fish fillet	0.74	0.15
55882	2011-724-062-1	Meramec River at Allenton Access	Golden Redhorse	Fish fillet	0.55	0.11
55883	2011-724-062-2	Meramec River at Allenton Access	Golden Redhorse	Fish fillet	0.81	0.16
55884	2011-724-062-3	Meramec River at Allenton Access	Golden Redhorse	Fish fillet	0.06	0.012
55885	2011-725-062-1	Meramec River at Route 66 State Park	Golden Redhorse	Fish fillet	0.61	0.12
55886	2011-725-062-2	Meramec River at Route 66 State Park	Golden Redhorse	Fish fillet	0.50	0.10
55887	2011-725-062-3	Meramec River at Route 66 State Park	Golden Redhorse	Fish fillet	0.47	0.093
55888	2011-724-420-1	Meramec River at Allenton Access	Longear Sunfish	Fish fillet	0.26	0.052
55889	2011-724-420-2	Meramec River at Allenton Access	Longear Sunfish	Fish fillet	0.48	0.10
55890	2011-724-420-3	Meramec River at Allenton Access	Longear Sunfish	Fish fillet	0.22	0.044
55891	2011-056-420-1	Big River at House Springs	Longear Sunfish	Fish fillet	0.16	0.032
55892	2011-056-420-2	Big River at House Springs	Longear Sunfish	Fish fillet	0.15	0.029
55893	2011-056-420-3	Big River at House Springs	Longear Sunfish	Fish fillet	0.12	0.025
55894	2011-459-230-1	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	1.14	0.23
55895	2011-459-230-2	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	0.41	0.081
55896	2011-459-230-3	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	1.30	0.26
55897	2011-514-230-1	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	0.17	0.034
55898	2011-514-230-2	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	0.32	0.064
55899	2011-514-230-3	Wakonda State Park at Agate Lake	Channel Catfish	Fish fillet	0.11	0.023
55900	2011-725-420-1	Meramec River at Route 66 State Park	Longear Sunfish	Fish fillet	0.29	0.058
55901	2011-725-420-2	Meramec River at Route 66 State Park	Longear Sunfish	Fish fillet	0.34	0.069
55902	2011-725-420-3	Meramec River at Route 66 State Park	Longear Sunfish	Fish fillet	0.30	0.060
55903	2011-025-406-1	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.15	0.030
55904	2011-025-406-2	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.28	0.055
55905	2011-025-406-3	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.46	0.093
55906	2011-025-406-4	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.46	0.092
55907	2011-025-406-5	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.30	0.060
55908	2011-025-406-6	Lake of the Ozarks at Niangua Arm	Largemouth Bass	Fish muscle plug	0.28	0.056
55909	2011-679-406-1	Longview Lake	Largemouth Bass	Fish muscle plug	1.13	0.23
55910	2011-679-406-2	Longview Lake	Largemouth Bass	Fish muscle plug	0.80	0.16

Table 4. Concentrations of total mercury in fish fillets, fish muscle plugs, and crayfish tissues.—Continued

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; Hg, mercury; µg/g, micrograms per gram; wet weight concentrations estimated assuming 80 percent moisture].

USGS ID	MDC ID	Site location	Biota common name	Matrix	Hg µg/g dry weight	Hg µg/g wet weight
55911	2011-679-406-3	Longview Lake	Largemouth Bass	Fish muscle plug	0.89	0.18
55912	2011-679-406-4	Longview Lake	Largemouth Bass	Fish muscle plug	1.50	0.30
55913	2011-679-406-5	Longview Lake	Largemouth Bass	Fish muscle plug	0.98	0.20
55914	2011-679-406-6	Longview Lake	Largemouth Bass	Fish muscle plug	0.98	0.20
55915	2011-118-406-1	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.19	0.038
55916	2011-118-406-2	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.26	0.053
55917	2011-118-406-3	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.43	0.086
55918	2011-118-406-4	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.28	0.057
55919	2011-118-406-5	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.13	0.027
55920	2011-118-406-6	Stockton Lake at Big Sac Arm	Largemouth Bass	Fish muscle plug	0.49	0.10
55921	2011-165-406-1	Clearwater Lake	Largemouth Bass	Fish muscle plug	2.84	0.57
55922	2011-165-406-2	Clearwater Lake	Largemouth Bass	Fish muscle plug	4.74	0.95
55923	2011-165-406-3	Clearwater Lake	Largemouth Bass	Fish muscle plug	2.48	0.50
55924	2011-165-406-4	Clearwater Lake	Largemouth Bass	Fish muscle plug	2.88	0.58
55925	2011-165-406-5	Clearwater Lake	Largemouth Bass	Fish muscle plug	1.76	0.35
55926	2011-165-406-6	Clearwater Lake	Largemouth Bass	Fish muscle plug	1.89	0.38
55927	2011-241-406-1	Lake St. Louis	Largemouth Bass	Fish muscle plug	1.83	0.37
55928	2011-241-406-2	Lake St. Louis	Largemouth Bass	Fish muscle plug	1.01	0.20
55929	2011-241-406-3	Lake St. Louis	Largemouth Bass	Fish muscle plug	0.75	0.15
55930	2011-241-406-4	Lake St. Louis	Largemouth Bass	Fish muscle plug	0.70	0.14
55931	2011-241-406-5	Lake St. Louis	Largemouth Bass	Fish muscle plug	0.44	0.088
55932	2011-241-406-6	Lake St. Louis	Largemouth Bass	Fish muscle plug	1.44	0.29
55933	2011-345-406-1	Noblett Lake	Largemouth Bass	Fish muscle plug	1.75	0.35
55934	2011-345-406-2	Noblett Lake	Largemouth Bass	Fish muscle plug	1.45	0.29
55935	2011-345-406-3	Noblett Lake	Largemouth Bass	Fish muscle plug	1.27	0.25
55936	2011-345-406-4	Noblett Lake	Largemouth Bass	Fish muscle plug	1.90	0.38
55937	2011-345-406-5	Noblett Lake	Largemouth Bass	Fish muscle plug	2.22	0.44
55938	2011-345-406-6	Noblett Lake	Largemouth Bass	Fish muscle plug	0.97	0.19
55939	2011-704-406-1	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	1.76	0.35
55940	2011-704-406-2	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	2.12	0.42
55941	2011-704-406-3	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	1.85	0.37
55942	2011-704-406-4	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	0.87	0.17
55943	2011-704-406-5	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	2.03	0.41

Table 4. Concentrations of total mercury in fish fillets, fish muscle plugs, and crayfish tissues.—Continued

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; Hg, mercury; µg/g, micrograms per gram; wet weight concentrations estimated assuming 80 percent moisture].

USGS ID	MDC ID	Site location	Biota common name	Matrix	Hg µg/g dry weight	Hg µg/g wet weight
55944	2011-704-406-6	Hazel Creek Lake	Largemouth Bass	Fish muscle plug	3.57	0.71
55945	2011-713-406-1	Harrison County Lake	Largemouth Bass	Fish muscle plug	1.49	0.30
55946	2011-713-406-2	Harrison County Lake	Largemouth Bass	Fish muscle plug	1.74	0.35
55947	2011-713-406-3	Harrison County Lake	Largemouth Bass	Fish muscle plug	1.47	0.29
55948	2011-713-406-4	Harrison County Lake	Largemouth Bass	Fish muscle plug	1.98	0.40
55949	2011-713-406-5	Harrison County Lake	Largemouth Bass	Fish muscle plug	3.34	0.67
55950	2011-713-406-6	Harrison County Lake	Largemouth Bass	Fish muscle plug	1.73	0.35
55952	2011-134-609-1-S	Big River at Cherokee Landing	Crayfish species	Carcass[a]	0.028	0.006
55954	2011-134-609-2-S	Big River at Cherokee Landing	Crayfish species	Carcass	0.037	0.007
55956	2011-134-609-3-S	Big River at Cherokee Landing	Crayfish species	Carcass	***0.017***	***0.003***
55958	2011-134-609-4-S	Big River at Cherokee Landing	Crayfish species	Carcass	0.026	0.005
55960	2011-134-609-5-S	Big River at Cherokee Landing	Crayfish species	Carcass	0.029	0.006
55962	2011-134-609-6-S	Big River at Cherokee Landing	Crayfish species	Carcass	0.024	0.005
55964	2011-134-609-7-S	Big River at Cherokee Landing	Crayfish species	Carcass	0.029	0.006
56227a	2011-134-609-M Composite[b]	Big River at Cherokee Landing	Crayfish species	Tail meat	0.052	0.010
55965	2011-726-609-1-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	0.023	0.005
55966	2011-726-609-1-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.008***	***0.002***
55967	2011-726-609-2-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	0.019	0.004
55968	2011-726-609-2-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.010***	***0.002***
55969	2011-726-609-3-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	0.027	0.005
55970	2011-726-609-3-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.011***	***0.002***
55971	2011-726-609-4-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	0.022	0.004
55972	2011-726-609-4-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.008***	***0.002***
55973	2011-726-609-5-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	0.029	0.006
55974	2011-726-609-5-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.013***	***0.003***
55975	2011-726-609-6-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	***0.011***	***0.002***
55976	2011-726-609-6-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.008***	***0.002***
55977	2011-726-609-7-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	***0.012***	***0.002***
55978	2011-726-609-7-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.006***	***0.001***
55979	2011-726-609-8-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	***0.011***	***0.002***
55980	2011-726-609-8-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.012***	***0.002***
55981	2011-726-609-8-T	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail shell	<0.005	<0.001
55982	2011-726-609-9-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	***0.008***	***0.002***

Table 4. Concentrations of total mercury in fish fillets, fish muscle plugs, and crayfish tissues.—Continued

[USGS, U.S. Geological Survey; MDC, Missouri Department of Conservation; Hg, mercury; µg/g, micrograms per gram; wet weight concentrations estimated assuming 80 percent moisture].

USGS ID	MDC ID	Site location	Biota common name	Matrix	Hg µg/g dry weight	Hg µg/g wet weight
55983	2011-726-609-9-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.012***	***0.002***
55984	2011-726-609-9-T	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail shell	<0.005	<0.001
55985	2011-726-609-10-M	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail meat	***0.007***	***0.001***
55986	2011-726-609-10-S	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Carcass	***0.010***	***0.002***
55987	2011-726-609-10-T	Shoal Creek 0.5 mile North of I-44 Bridge	Crayfish species	Tail shell	<0.005	<0.001

[a]Carcass is defined as crayfish remains after tail meat has been removed or extracted.

[b]Because of insufficient biomass in submitted individual samples, this sample is a composite of seven crayfish tail meat samples collected from the Big River at Cherokee Landing (55951, 55953, 55955, 55957, 55959, 55961, 55963).

levels were markedly higher in crayfish carcass samples from the Big River at Cherokee Landing (3 to 7.8 µg/g dry weight; USGS IDs 55952-55964) and tail-meat levels were most elevated in the tail meat composite sample from this site (1.06 µg/g dry weight; USGS ID 56227). Cadmium concentrations (µg/g dry weight) in crayfish tissues collected from the one remaining site (Shoal Creek; USGS IDs 55965-55987) ranged as follows: carcass, 0.2 to 0.5; tail shell, 0.02 to 0.042; and tail meat, 0.017 to 0.33. Lead concentrations (µg/g dry weight) in fish fillet samples mostly were less than 0.5 (76 percent of the sample set), whereas remaining samples ranged from 0.81 to 1.5. All crayfish tissues from Shoal Creek exhibited Pb concentrations ranging from 0.17 to 4.74 µg/g dry weight. This is in sharp contrast to Pb concentrations in crayfish carcass samples and a tail meat composite in crayfish collected from Cherokee Landing (21.9 to 44.9 µg/g dry weight).

All fish fillet samples exhibited Hg concentrations less than the current (2009) U. S. Environmental Protection Agency (USEPA) Water Quality Criterion for Hg of 0.30 µg/g wet weight (table 4), or about 1.5 µg/g dry weight based on an 80 percent moisture content (U. S. Environmental Protection Agency, 2001); however, a number of dorsal fish muscle plugs collected from largemouth bass exceeded this criterion, and the site, number of plugs, and Hg concentration ranges (µg/g dry weight) were as follows: Clearwater Lake, 6 plugs, 1.76 to 4.74; Lake St. Louis, 1 plug, 1.83; Noblett Lake, 3 plugs, 1.75 to 2.22; Hazel Creek Lake, 5 plugs, 1.76 to 3.57; Harrison County Lake, 4 plugs, 1.73 to 3.34. Mercury concentrations in all crayfish tissues were quite low (less than 0.06 µg/g dry weight).

Quality-Control Results

Calibration Verification.—A calibration blank and an independent calibration verification standard (ICVS) were analyzed every 10 samples to confirm the calibration status of the ICP-MS during instrumental analyses of the fish fillets, fish muscle plugs, and crayfish tissues for Ca, Zn, Cd, and Pb; blanks were within plus or minus (±)three times the instrument detection limits for each element, and ICVS recoveries were within the target of 90 to 110 percent of the ICVS standard concentration for each element. Three reference solutions [National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1640a: Trace Elements in Natural Water; Spex ClaritasPPT Instrument Check Standard 1 (Spex ICS-1); and High Purity Standards Certified Reference Solution Trace Metals in Fish: HP CRM-TF] used as laboratory-control samples exhibited elemental recoveries ranging from 98 to 100 percent. Calibration verification reference tissues for total Hg [National Research Council Canada (NRCC) DOLT-3: Dogfish Liver; International Atomic Energy Agency (IAEA) 407-Trace Elements and Methylmercury in Fish Tissue] were analyzed at the beginning and end of

the instrumental runs to confirm the calibration status of the DMA-80 system; percent errors were within the target of ±10 percent.

Reference Materials.—Recoveries of Ca, Zn, Cd and Pb in three tissue reference materials (NIST 1566a: Oyster Tissue, n=1; IAEA 407: Whole-body Fish, n=1; NRCC TORT-1: Lobster Hepatopancreas, n=1) ranged from 80 to 120 percent and averaged 100 percent. Recoveries of Hg from six different tissue reference materials (IAEA MA-A-1: Copepod, n=4; IAEA MA-A-2: Fish Flesh Homogenate, n=4; IAEA 407: Whole-body Fish, n=4; NIST RM50: Albacore Tuna, n=1; NRCC DOLT-3: Dogfish Liver, n=4; and NRCC DORM-2: Dogfish Fillet, n=4;) ranged from 89 to 105 percent and averaged 98 percent.

Method and Instrumental Precision.— Method precision as estimated from measurement variation for triplicate digestion and analysis of a fish fillet sample had percent relative standard deviations (PRSDs) for Ca, Zn, and Cd that were less than or equal to (≤) 26, but 60 for Pb; however, the higher variation for Pb was not considered unusual because two of the three sample concentrations were below the method quantitation limit of 0.026 µg/g dry weight. PRSDs for the elements from a crayfish carcass replicate were ≤ 10. Relative percent differences (RPDs) from the duplicate digestion and analysis of the crayfish tail meat composite sample were ≤ 21 for Ca, Zn, and Pb, but higher (40) for Cd. All PRSDs for Hg (n=4) were less than (<) 9 percent. Instrumental precision measured as RPD from the analysis of fish fillet (n= 3) and crayfish tail meat (n=3) duplicate digestates for Ca, Zn, Cd, and Pb was < 5 percent.

Spikes.— Recoveries of Zn, Cd, and Pb spiked into fish tissue (n=2) ranged from 105 to 107 percent and averaged 106 percent, whereas Ca spike recoveries were 111 and 145 percent. Recoveries of all four elements in spiked crayfish tissues (n=2) ranged from 103 to 120 percent and averaged 111 percent. Recoveries of methylmercury hydroxide spiked into fish fillet (n=2), fish muscle plugs (n=4), and crayfish tissue (n=2) ranged from 70 to 101 percent and averaged 88 percent. Post-digestion or analysis spikes for Ca, Zn, Cd, and Pb in fish fillets (n=3) and crayfish tissues had recoveries ranging from 81 to 105 percent and averaged 96 percent.

Interference Checks.— As a check for potential interferences, dilution percent differences (DPDs) based on fivefold dilutions of fish fillet (n=3) and crayfish tail-meat digestates were determined. DPDs were ≤ 11 percent for Zn, Cd, and Pb, but higher for Ca (14 to 27 percent), suggesting some suppression of the Ca signal in these matrices. A synthetic solution containing high concentrations of aluminum, Ca, iron, magnesium, sodium, phosphorus, potassium, sulfur, carbon, molybdenum, and titanium was analyzed (n=4) to observe the effects of these potential interfering elements on the determination of Zn, Cd, and Pb concentrations in this matrix. Recoveries were within the 80 to 120 percent tolerance

Blank Equivalent Concentrations (BEC).— BECs (µg/g dry weight, Ca, Zn, Cd, Pb) for digestion blanks prepared with each batch were determined; all BECs were less than the corresponding method detection limits (MDLs) except for one instance of Zn (0.16 BEC compared to 0.11 µg/g MDL), one instance of Cd (0.002 BEC compared to 0.002 µg/g MDL), and one instance of Pb (0.009 BEC compared to 0.007µg/g MDL). The BEC exceptions each represent low concentrations that were not considered unusual. All BECs for Hg were less than the corresponding Hg MDLs

Instrument Detection, Method Detection, and Method Quantitation Limits.—The instrument detection limit (IDL) for Hg was 0.002 nanograms (ng); the IDLs for other target analytes in nanograms per milliliter were as follows: Ca, 2.3; Zn, 0.9; Cd, 0.003; and Pb, 0.004. MDLs were computed in µg/g dry weight for each batch of samples as:

$$3 \text{ X } (SD_b^2 + SD_s^2)^{1/2}$$

where

SD_b = standard deviation of a blank (n=3); and
SD_s = standard deviation of a low level sample or spiked sample (n=3)

and were as follows:Ca, 1.6 to 2.9; Zn, 0.03 to 0.6; Cd, 0.002 to 0.012; Hg, 0.005 to 0.042; and Pb, 0.007 to 0.009. Method quantitation limits (MQLs) were calculated in µg/g dry weight as 3.3 x MDLs and were as follows: Ca, 5.3 to 9.6; Zn, 0.1 to 2.0; Cd, 0.007 to 0.040; Hg, 0.018 to 0.14; and Pb, 0.023 to 0.030.

All quality-control results for the study were within acceptable limits as specified by USGS.

References Cited

Schmitt, C.J., and Finger, S.E., 1987, The effects of sample preparation on measured concentrations of eight elements in edible tissues of fish from streams contaminated by lead mining: Archives of Environmental Contamination and Toxicology, v.16, p.185-207.

U. S. Environmental Protection Agency, 1998, Mercury in solids and solutions by thermal decomposition, amalgamation, and atomic absorption spectrophotometry: accessed August 28, 2007, at *http://www.epa.gov/epaoswer/hazwaste/test/up4a.htm#7_series*.

United States Environmental Protection Agency, 2001, Water quality criterion for protection of human health - Methylmercury: Washington D.C., U. S. Environmental Protection Agency, Office of Water, EPA-823-R-01-001, 16 p.

Publishing support provided by:
Rolla Publishing Service Center

For more information concerning this publication, contact:
Director, USGS Columbia Environmental Research Center
4200 New Haven Road
Columbia, MO 65201
(573) 875–5399

Or visit the Columbia Environmental Research Center Web site at:
http://www.cerc.usgs.gov/